



THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of :

Applicant : Masahiko HIROSE, et al.

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Title : COMPOSITE REVERSE OSMOSIS MEMBRANE AND METHOD  
FOR PRODUCING THE SAME

DECLARATION UNDER RULE 37 CFR 1.132

HON. COMMISSIONER FOR PATENTS AND TRADEMARKS  
WASHINGTON, D.C. 20231

SIR:

I, Masahiko HIROSE, hereby declare that I reside at 203-11, Ryousenji, Ritto-cho, Kurita-gun, Shiga, Japan, and I am a citizen and resident of Japan, and hereby further declare as follows:

I was born July 5, 1960, and graduated from Kyoto University, department of polymer chemistry, in March 1984. I then joined Nitto Denko Corporation, and have been engaged in the development of the reverse osmosis membrane to the present. I am the named inventor for the above application.

The following describes experiments that were carried out under my supervision.

Object of the experiment

The object of the experiments is to demonstrate the superiority of the present invention over the cited references, USP 5,178,766 and USP 6,171,497.

## Experiments

### 1) Superiority of the present invention over USP 5,178,766

#### (Experimental Conditions)

Samples of a composite reverse osmosis membrane were produced according to the process disclosed in Example 1 and Comparative Example 1 of the present invention. Then, the performance of both the sample membranes was evaluated under the test conditions of USP 5,178,766, specifically, using a 1500 ppm NaCl solution having a pH of 6.5 at an operation pressure of 15 kgf/cm<sup>2</sup> and a temperature of 25°C.

#### (Result)

The results of the above experiment are shown in the following Table along with the test conditions and results in the present invention.

Sample	Test condition applied	Test Conditions				Performance	
		NaCl concentration (ppm)	Operation pressure (kgf/cm <sup>2</sup> )	Temperature (°C)	pH	NaCl rejection (%)	Flux (m <sup>3</sup> /m <sup>2</sup> · day)
Membrane of Ex. 1	Present invention	500	5	25	6.5	99.5	1.1
	USP '766	1500	15	25	6.5	99.6	3.3
Membrane of Com. Ex. 1	Present invention	500	5	25	6.5	93	0.7
	USP '766	1500	15	25	6.5	97	2.1

#### (Consideration)

As can be seen from the above Table, under the test conditions of USP 5,178,766, the membrane of Example 1 of the present invention achieved a NaCl rejection of 99.6%. In contrast, the NaCl rejection of the composite reverse osmosis membrane according to Example 1 of USP 5,178,766 is 98.1%, which is much lower than the NaCl rejection of the membrane of the present invention and is actually at the similar level as that of the sample membrane according to Comparative Example 1 of the present invention, which achieved a NaCl rejection of 97% under the test conditions of USP 5,178,766.

Therefore, it is apparent that the performance of the composite

reverse osmosis membrane disclosed in USP 5,178,766 is at the level of the prior art disclosed in the present invention.

## 2) Superiority of the present invention over USP 6,171,497

### (Experimental Conditions)

A sample of a reverse osmosis membrane was produced in the same manner as in Example 1 of USP 6,171,497, and the performance thereof was evaluated using a 500 ppm NaCl solution having a pH of 6.5 at an operation pressure of 5 kgf/cm<sup>2</sup> and a temperature of 25°C. The performance of the sample membrane also was evaluated under the same test conditions as described above except that 500 ppm isopropyl alcohol was used as a solute instead of NaCl.

Furthermore, a sample of a composite reverse osmosis membrane was produced according to the process disclosed in Example 1 of the present invention, and the performance thereof was evaluated using a solution containing 500 ppm isopropyl alcohol as a solute and having a pH of 6.5 at an operation pressure of 5 kgf/cm<sup>2</sup> and a temperature of 25°C.

### (Result)

As a result, the sample according to Example 1 of USP 6,171,497 achieved a NaCl rejection of 99.3% and a flux of 1.0 m<sup>3</sup>/m<sup>2</sup>·day and an isopropyl alcohol rejection of 58%.

On the other hand, the sample membrane according to Example 1 of the present invention achieved an isopropyl alcohol rejection of 75%.

### (Consideration)

With regard to NaCl as a charged salt, the rejection achieved by the membrane according to the present invention does not differ considerably from that achieved by the membrane according to USP 6,171,497. However, with regard to isopropyl alcohol as a non-charged solute, the rejection achieved by the membrane according to the present invention differs from that achieved by the membrane according to USP 6,171,497 by 17 points. That is, the membrane according to the present invention achieves the rejection about 20% higher than that of the membrane according to USP 6,171,497.

The difference in isopropyl alcohol rejection is caused by the difference in the process for forming membrane between USP 6,171,497 and

the present invention. Specifically, in USP 6,171,497, a membrane with a large specific surface area is treated with an oxidizing agent to further degrade a portion of the surface thereof. Although such a reverse osmosis membrane can achieve a high rejection ratio for a charged salt due to the effect of the repulsion of charged particles, it cannot achieve a high rejection ratio for a non-charged solute such as organic substances because the non-charged solute can pass through the membrane from the portion degraded by the oxidizing agent on the surface of the membrane.

In contrast, in the present invention, the hydrophilicity of the surface of the membrane is improved without degrading or causing a defect on the surface of the membrane. Therefore, the reverse osmosis membrane according to the present invention can achieve a high salt rejection ratio without decreasing a rejection ratio for organic substances.

I, the undersigned declarant, declare further that all statements made herein are true and that all statements made are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001, of Title 18, of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this July 16 , 2003, at Osaka, JAPAN

Masahiko Hirose

Masahiko HIROSE